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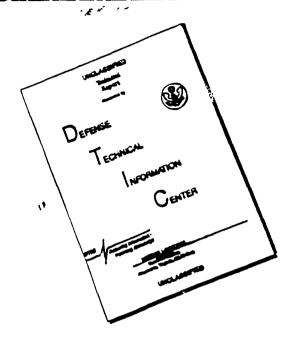
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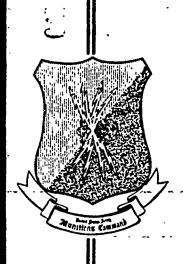


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TECHNICAL MEMORANDUM 1149

COMPOSITION B4,
A NON-EXUDING EXPLOSIVE FILLER

FOR

ARTILLERY SHELL:

MORRIS E. POLLACK
ROBERT T. SCHIMMEL
STANLEY J. LOWELL

COPY NO. 21 OF 42.

APRIL 1963

PICATINNY ARSENAL DOVER, NEW JERSEY

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ARTILLERY SHELL

BY

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(/)) APRIL 1963,

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OBJECT

Composition B4 is to be established as the explosive filler replacement for Composition B in cast-loading artillery shell.

SUMMARY

Composition B4 (60/39.5/0.5 RDX/TNT/calcium silicate), a non-exuding explosive, was satisfactorily loaded into Shell, HE, 175mm T203E9. Laboratory test results indicated that this non-exuding explosive is less sensitive to impact than Composition B. Safety tests have been conducted on 200 Composition B4-loaded T203E9 175mm Shell. These were fired at chamber pressures from 105-120% of gun rated pressure and satisfactorily withstood setback pressures of 27,000 psi.

CONCLUSION

Although shell loaded with Composition B4 develop more cracks than those with Composition B they can be fired satisfactorily in all of the currently standard artillery weapons. The sensitivity of RDX/TNT compositions is not adversely affected by ommission of the wax, so-called, desensitizing agent.

RECOMMENDATION

Composition B4 should be substituted for Composition B as the standard explosive filler for ammunition.

INTRODUCTION

The exudation of TNT and TNT-containing explosives at elevated temperatures has been a serious problem in ammunition storage over the years. Explosives engineers and ammunition designers have faced this problem and proposed several methods to minimize or eliminate exudation in artillery ammunition.

Initially, sealants and gaskets were used to retard the flow of exudate from the explosive charge into fuze threads of an artillery projectile. These were ineffective. As reported in Reference 1, calcium silicate was successfully used to prevent the flow of exudate into shell fuze threads of the M71 90mm Shell. Finely powdered calcium silicate possesses a high absorbing capability and is easy to apply as a packing at the bottom of the fuze well of an artillery projectile.

Reference 2 recommended in February 1960, that whenever possible, the addition of calcium silicate absorbent to TNT-containing explosives should be adopted for all ammunition items such as mines and certain warheads that were not subjected to high acceleration or deceleration forces. This recommendation came after concluding that calcium silicate, to 1.25% of the TNT content, reliably prevents exudation in certain warheads. A laboratory study was conducted to determine whether the sensitivity of Composition B was affected by the removal of the desensitizing wax and the addition of calcium silicate. It was concluded that the effectiveness of Composition B is increased, the sensitivity is unaffected and the elimination of exudation is assured by the removal of the desensitizing wax and the substitution of calcium silicate (Reference 3).

Another problem of concern to the explosives engineer over the years is that of cracks in the explosive filler. Military Specification MIL-L-20336A dated 14 December 1954 (Appendix C) permits no more than two cracks in the explosive charge. Although this specification has been the standard since 1954 for loading experimental and production artillery rounds, it has been too severe as the acceptance criteria (Reference 3). A recent loading specification for M470 155mm HE Shell (MIL-L-46633A, 30 November 62) permits not more than four transverse cracks in any charge and not more than one transverse crack in Segment A.

An explosive mixture of Composition B and 0.5% calcium silicate was cast-loaded into 394 T387E1 155mm Shell for a safety test. Initial radiographic examination indicated that the shell filler contained an excessive number of cracks. However, the shell were accepted and shipped to Aberdeen Proving Ground, Maryland, for safety firing tests. A second radiographic examination made at the Proving Ground, prior to firing, indicated there were additional cracks as a result of jolting the loaded shell received during transportation.

All of these shell and the XM51E2 Propelling Charge were conditioned and fired at 140°F to produce a chamber pressure of 49,200 psi in the T258 Howitzer. This was 117% of service pressure. All of the shell fired satisfactorily (Reference 5). Since such promising results were obtained for Composition B with 0.5% calcium silicate added, this investigation is to ascertain whether Composition B4, an explosive similar to Composition B with 0.5% calcium silicate (except the 1% desensitizing wax is eliminated) can replace the currently standard Composition B, even though the Composition B4 is more crack prone.

RESULTS

Two hundred T203E9 Shell were loaded with Composition B4 and supplementary charges in July 1960 for testing (in connection with Test Program Request TE-244 dated 13 April 1960). Details of the test are described in Aberdeen Proving Ground Firing Record 68019, which covers tests conducted from 29 March-18 April 1962 (Reference 4). These shell were fired, without incident, at temperatures of 70 and 140 F.

The characteristics and properties of Composition B4 are summarized in Table I.

TABLE I

Properties of Composition B4 and Composition B

	Comp B4	Comp B
Sensitivity to Impact		
PA Apparatus 2 Kg wt, inches PA Apparatus, 2 Kg wt, 18 in, drop height, no. reactions in 50 tests	16 18	14 3 1
Explosion Temperature Test	272°C	278°C
No flame, flash or explosion smoke evolved in 5 sec		gecomboses
Brisance		
Sand Crushed	56.6	54.0
arge (Lead Azide) ing Charge (Lead Azide)	0.300	0,200
Density gm/ml (20 C)	1,73	1.68
Hygroscopicity at 20°C and 95% RH, at 30°C and 90% RH	0,16	0.02
Viscosity, efflux at 85°C rec.	4 n n n o 4	

TABLE I (Continued)

	Comp B4	Comp B
100°C Vacuum Stability Test (40 hr) ml gas	0.17	0.70
120°C Vacuum Stability Test (40 hr) ml gas	0.61	06.0
Rate of Detonation d = 1.71 g/ml m/sec d = 1.68 g/ml m/s	7,800	7,840
Friction Pendulum Test		
Steel Shoe	Unaffected	Unaffected

TABLE I (Continued)

	Comp B4	Comp B
100°C Vacuuri Stability Test (40 hr) ml ga	6.17	0.73
120°C Vacuum Stability Test (40 hr) ml gas	0,61	0.90
Rate of Detonation d = 1,71 g/ml m/sec d = 1,68 g/ml m/s	7,800	7,840
Friction Pendulum Test		
Steel Shoe	Unaffected	Unaffected

DISCUSSION OF RESULTS

The desensitizing effect of wax on Composition B has been questioned many times since its adoption as an ingredient in the explosive composition. During the development of Composition B4 as a substitute for Composition B, the impact and friction sensitivities of the two explosive fillers were compared. Table I indicates there does not appear to be any significant difference between Composition B and Composition B4 with respect to impact and friction sensitivity. To confirm the fact that there is little difference in impact sensitivity between the two explosives, a modified impact test was conducted using the standard Picatinny Arsenal Test Apparatus. A drop height of 18 inches was selected for the impact test because sufficient reactions occurred with both explosive compositions at this height, to allow for a comparison of their relative sensitivity. Fifty samples each of Composition B and Composition B4 were subjected to the modified test. Reactions occurred in 31 of 50 samples of Composition B and in 18 of 50 samples of Composition B4. This indicated that wax had no apparent desensitizing effect on the Composition B. The elimination of the so-called desensitizing wax from Composition B and the addition of 0.5% calcium silicate as an absorbent does not adversely affect the impact sensitivity. The rates of detonation for Composition B and Composition B4 compare favorably (Table I). This indicates that Composition B4 may be expected to perform at least as well as Composition B. Since the 1% wax is to be eliminated and only 0.5% calcium sili ate is to be added, the explosive content should rise 0.5%. This should increase the performance potential of Composition B4.

It is well established that TNT-containing explosives exude at elevated temperatures. This occurrence has been investigated frequently over the years. Reference 1, 2 and 3 report the latest accomplishments at Picatinny Arsenal in this area. Calcium silicate was recommended as the absorbent that will minimize the tendency for TNT-containing explosives to exude. Firing tests of T203E9 175mm HE Shell, loaded with Composition B4, indicated that this explosive composition can be fired safely in the T145E4 175mm Gun at all temperatures between -40 F and 140 F. Since this weapon is the highest setback force artillery weapon, it is reasonable to assume that all shell loaded with Composition B4 can be safely fired in weapons that do not exceed the setback pressure of 27,000 psi encountered in the firing tests.

There has been concern over the number of cracks that develop in artillery shell cast-loaded with Composition B4 (Appendix A and B). This concern may be attributed to the strict requirements in Specification MIL-L20336A (Appendix C). The requirements in this specification have been the acceptance criteria for shell cast-loaded with Composition B and TNT for a long time. The specification allows for no more than two transverse cracks in any charge and not more than one crack in Segment A. Reference 6 cites the experience of Picatinny Arsenal and the Iowa Ordnance Plant in loading shell with RDX/TNT compositions.

The experience gained in loading the T387E1 155mm Shell with 99.5/0.5 Composition B/calcium silicate is of special interest since it reemphasizes that Specification MIL-L-20336A is, in some cases, too rigid. After successfully testing the T387E1 (M470) 155mm Shell, the specification for loading the shell (MIL-P-46633A dated 30 November 1962) allowed four transverse cracks rather than the two permitted by Specification MIL-L20336A. This is a clear departure from the former rigid acceptance standards established to assure cast-loading of acceptable quality. Indications are there will be a further relaxation in the requirements allowing for more cracks in cast-loaded shell. This should allay the concern regarding acceptance of shell with cracks that appear in segments other than A. Current safety tests will furnish additional data to indicate whether or not confidence in firing shell with multiple cracked charges is justified.

REFERENCES

- 1. R.W. Heinemann and C.J. Horvat, Control of Exudation in the 90mm
 M71 Loaded Shell, Picatinny Arsenal Technical Report 2463, December
 1957.
- 2. R.W. Heinemann and S.J. Lowell, <u>Prevention of Exudation from Ammunition Item</u>, Picatinny Arsenal Technical Report 2675, February 1960.
- 3. R.T. Schimmel and R.J. Heredia, Stability of H6, Composition B and Modifications of Same, Initiator and Explosives Application Section, Artillery Ammunition and Rocket Development Laboratory Report 100, October 1961.
- 4. SP/4 L.R. Douschug, Report on Shell, 175mm, T203E9, Composition B4-Loaded, Development and Proof Services, Aberdeen Proving Ground Report DPS-625, August 1962.
- 5. J. Doyle and M. Goldstein, STReview Point Notes for Projectile, 155mm HE T387El and Charge, Propelling XM51El (U), Artillery Ammunition and Rocket Development Laboratory, Picatinny Arsenal, November 1961.
- 6. R.T. Schimmel, Acceptance Criteria for Composition B/Calcium
 Silicate Loaded 155MM T387E1 Shell Initiator and Explosives Application Section, Artillery Ammunition and Rocket Development Laboratory Report No. DR4-56, December 1961.

APPENDICES

APPENDIX A

Military Sepcification MIL-C-46652 (Ord) Composition B4

MILITARY SPECIFICATION

COMPOSITION B4

1. SCOPE

1.1 This specification covers one type of high explosive designated as Composition B4 for use in Ammunition.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieves; Standards, For Testing Purposes.

MILITARY

MIL-R-00398 - RDX.

JAN-T-248 - Trinitrotoluene (TNT) MIL-C-51077 - Calcium Silicate, Technical.

STANDARDS

FEDERAL

Federal Test Method-STD-791 - Lubricants, Liquid Fuels, and Related Products. Methods of Inspection, Sampling, and Testing.

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.

MIL-STD-109 - Inspection Terms and Definitions.

FSC: 1375

DRAWINGS

ORDNANCE CORPS

7548644 - Box, Packing for High Explosives, Assembly

Details, Packing and Marking.

7548645 - Carton, Packing Reusable-Collapsible for High Explosives., Assembly, Details, Packing and Marking.
81-3-148 - Efflux Viscosimeter for Explosives.

PUBLICATIONS

ORDNANCE CORPS

ORD-M608-11 - Procedures and Tables for Continuous Sampling by Attributes.

(Copies of specifications, standards, drawings and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer).

3. REQUIREMENTS

3.1 Material.-The constituent materials used in the manufacture of Composition B4 shall comply with the following specifications:

Constituent Material	Conforming to:		
RDX	MIL-R-00398		
Trinitrotoluene (TNT)	Type B JAN-T-248		
	Grade I		
Calcium Silicate	MIL-C-51077		

3.2 Composition.-The composition of Composition B4 shall be as specified in Table I when tested as specified in 4.3.1.

RDX, percent	60.0 plus or minus 2.0
TNT, percent	39.5 plus or minus 2.0
Calcium S'licate	0.5 plus or minus 0.1

- 3.3 Moisture.-The moisture content shall be 0.25 percent maximum (max.) where recalled a specified in 4.3.2.
- 3.4 Viscosity.-The viscosity shall be 7.0 efflux seconds, max., when tested as specified in 4.3.3.
- 3.5 Insoluble particles.-Not more than five particles shall te retained on a number 60 United States Standard (US) sieve when tested as specified in 4.3.4.
- 3.5 Form.-Unless otherwise specified in the contract or purchase order, Composition B4 shall be supplied in the form of buds, or as strips approximately 1 1/2 inches wide and 3 inches long when tested as specified in 4.3.5.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 General quality assurance provisions.—The supplier is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements. Reference shall be made to Standard MIL—STD—109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.
- 4.1.1 Contractor quality assurance system.—If the contractor desires to utilize a quality assurance system, which is at variance with the quality assurance provisions of 4.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection, (measuring and testing equipment), and provisions for control and disposition of non-conforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance provisions required by the provisions of 4.2 and 4.3 and the other documents referenced herein. The contractor shall not be restricted to the inspection station

nor the method of inspection listed in this specification provided that an equita and control is included in the approved quality assurance procedure. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any changes to the written procedure which effects the degree of assurance required by this specification or other documents referenced herein.

- 4.1.2. Submission of product.—At the time the completed lot of product is submitted to the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product being submitted:
- a. A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.
 - b. Quantity of product inspected.
 - c. Results obtained for all inspection performed.
- d. Specification number and date, together with an identification and date of changes.
- e. Certificates of analysis on all material procured directly by the contractor when such material is controlled by Government specifications referenced in any of the contractual documents.
 - f. Quantity of product in the lot.
- g. Date submitted.
 The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.
- 4.1.3 Government verification.-Using the contractor's written quality assurance procedure (see 4.1.1) this detail specification, and other contractual documents as a guide, the Government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with a. or b. as applicable, the decision being the responsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality provisions of other specifications

referenced in any of the contractual documents have been complied with. Deviations in magnitude or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be as directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejection, screening, resampling, re-instruction of the supplier's employees, or other appropriate action:

a. Verification at the point of manufacture shall be accomplished at unscreduled intervals in accordance with

4.1.3.1 and 4.1.3.2.

b. Verification at the point of delivery shall be in accordance with 4.1.3.2.

- 4.1.3.1 Surveillance.-Surveillance shall include, but is not limited to:
- a. Observation of procedures concerning lot formation and identification.
- b. Observation of sampling procedures and application of acceptance criteria.
- c. Determination that all required examinations and tests are performed in accordance with the prescribed procedures of this specification, or approved equivalents thereto.
- d. Review of procedures for control and disposition of non-conforming material.
- 4.1.3.2 Product inspection.-Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection by the Government shall be performed to the degree necessary in order to determine that the product is of the quality required by this specification and that the contractor's records are reliable.

4.2 Inspection provisions.

4.2.1 Lot formation.-A lot shall consist of one or more batches of Composition B4 produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each batch shall consist of that quantity of Composition B4 that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous.

- 4.2.2 Examination.-Sampling plans and procedures for the following classification of defects shall be in accordance with Standard MIL-STD-105. Continuous sampling plans, in accordance with Handbook ORD-M608-11 may be used if approved by the procuring activity. Also, at the option of the procuring activity, AQL's and sampling plans may be applied to the individual characterisites listed using an AQL of 0.25 percent for each major defect and an AQL of 0.40 percent for each minor defect.
- 4.2.2.1 Wooden box or fiberboard carton, prior to closing (see dwg. F7548644, and F7548645,)

Categorio		Method of Inspection	
Critical	: None defined .		
102.	AQL 0.40 percent Liner pierced or torn Liner improperly closed Foreign matter	Visual	01001 01002 01003
Minor: 201.	AQL 0.40 percent Type of liner incorrect	Visual	01004
4.2.2.2	Sealed wooden box (see dwg. F75	48644).	i
Categori		Method of Inspection	Code No.
Critical	None defined	٠.	
102. 103.	Box damaged	Visual Visual	02001 02002 02003 02004
Minor: 201. 202. 203.	AQL 1.50 percent Nail protruding Marking misleading or unidentifiable Strapping improperly assembled.	Visual	02005 02006 02007

4.2.2.3 Sealed fiberboard carton (see dwg. F7548645)

Categories Defects Method of Code No.
Inspection

Critical: None defined

Major:	AQL 0.40 percent		
	Assembly torn or pierced	Visual	03001
102.	DOD symbol misleading or		
	unidentifiable		03002
103.	Strapping broken or loose	Visual-	
		Manual	03003
103.		Visual-	03003

- 4.2.3 Testing.-Samples shall be selected from each lot of Composition B4, in such number and amount as to insure that the samples shall be representative of the lot and shall be subjected to all tests specified herein. If the sample fails to comply with any of the requirements specified herein, the lot shall be rejected. The tests shall be performed as specified in 4.3.
 - 4.3 Test methods.
 - 4.3.1 Composition.
- 4.3.1.1 (TNT) content, Code No. 04001.-A weighed portion of 5.00 grams (gms.) of the sample shall be placed in a 500 milliliter (ml.) beaker, and 15 ml. of benzene saturated with RDX shall be added. The beaker shall be covered-with a watch glass and placed on a steam bath for 30 minutes. The lumps shall be broken up with a glass rod and the solution agitated occasionally by swirling. After cooling to room temperature the solution shall be filtered through a tared filtering crucible. The insoluble residue shall be transferred from the beaker to the filtering crucible using four portions or more of 2 to 3 ml. each of benzene saturated with RDX. Air shall be drawn through the crucible until the odor of benzene is no longer detectable. The crucible and contents shall be dried for one hour at 100 degrees Centigrade (C.) plus or minus 5 degrees C., cooled in a desiccator, and weighed. The percentage of TNT shall be calculated as follows:

Percent TNT = $\frac{100 \text{ A-B}}{\text{W} - (\text{MW})}$

where:

A = weight of crucible and sample.

B = weight of crucible and residue after benzene extraction.

W = weight of sample.

M = percent moisture in material, expressed as a desimal (see 4.3.2).

4.3.1.2 RDX content, Code No. 05001.-The crucible and residue from the TNT determination shall be extracted with 8 ten ml. portions of hot acetone. The acetone shall be allowed to remain in contact with the sample for one minute before applying suction. Air shall be drawn through the crucible until the odor of acetone is no longer detectable. The crucible and contents shall be dried for one hour at 100 degrees C. plus or minus 5 degrees C. cooled_in a desiccator, and weighed. The percentage of RDX shall be calculated as follows:

Percent RDX = 100 B-C

where:

B = weight of crucible and residue after benzene extraction. C = weight of crucible and residue after acetone extraction.

W = weight of sample.

M = percent moisture in material, expressed as a decimal (see 4.3.2).

4.3.1.3 Calcium silicate, Code No. 06001.-The percent of calcium silicate shall be calculated as follows:

Percent Calcium Silicate = 100 C-D w - (MW)

where:

C = weight of crucible and residue after acetone extraction.
D = weight of crucible and residue.
W = weight of sample.

M = percent moisture in material, expressed as a decimal (see 4.3.2).

4.3.1.4 Alternate method for TNT, Code No. 04002.-An accurately weighed portion of exactly 1.000 gm. of sample shall be transferred to 100 ml. volumetric flask and add 60

8

ml. of benzene (previously saturated with RDX). The flask shall be loosely stoppered and placed on a steam bath for 5 to 10 minutes with occasional swirling. The flask shall te removed from the steam bath and cooled to room temperature. The flask shall be made up to the mark with benzene saturated with RDX and shaken well and allow the RDX to settle to the bottom. Twenty ml. of the clear supernatant solution shall be transferred, using a pipet, to a 500 ml. titration flask from which the air has been swept out by a stream of carbon dioxide gas. Continue the flow of gas throughout the determination. Twenty five ml. of acetic acid and 25 ml. of concentrated hydrochloric acid shall be added to the titration flask and the contents shall be stirred by means of a magnetic stirrer for 10 minutes. Exactly 100 ml. of 0.2 normal (N) chromous chloride solution (see 6.3) shall be added to the flask and again stirred for 10 minutes. Fifteen drops of 2 percent aqueous phenosafranin indicator shall be added and titrated with 0.15 N ferric ammonium sulfate solution until a sharp color change from green to deep red. A blank determination shall be run following the same procedure but omit the sample. A constant temperature must be maintained for the blank and the sample. The percent of TNT shall be calculated as follows:

Percent TNT =
$$6.310 \text{ N } (V_3 - V_4)$$

W - (MW)

where:

 V_4 = ml. of ferric ammonium sulfate used to titrate the sample.

V₃ = ml. of ferric ammonium sulfate used to titrate the blank.

N = normality of the ferric ammonium sulfate solution.

W = weight in gm.

M = percent moisture in material, expressed as a decimal (see 4.3.2).

4.3.1.5 Alternate method for RDX, Code No. 05002.—An accurately weighed portion of exactly 0.5000 gm. of the sample shall be transferred to a 100 ml. volumetric flask. About 60 ml. of acetic acid shall be added to the flask and heated on a hot plate until the sample is completely dissolved. The flask shall be removed from the hot plate and allow to cool to room temperature. The flask shall be made up to the mark with acetic acid and shaken. Twenty ml. of the contents of the flask shall be transferred, using a pipet, to a 500 ml. titration flask from which the air has been swept out by a stream of carbon dioxide gas. Continue the flow of gas throughout the determination. Twenty five ml. of concentrated hydrochloric acid shall be added and stirred with a magnetic stirrer for 10 minutes. Exactly 100 ml. of 0.2 N chromous

chloride solution shall be added to the flask and stirred for 15 minutes. Fifteen drops of phenosafranin indicator shall be added and titrate with 0.15N ferric ammonium sulfate solution until a sharp color change from green to deep red. A blank shall be run following the same procedure but omit the sample. The percentage of RDX shall be calculated as follows:

Percent RDX = 3.085 N
$$(v_1 - v_2) - (v_3 - v_4)$$

where:

V₁ = ml. of ferric ammonium sulfate used to titrate the blank.

V2 = ml. of ferr'c ammonium sulfate used to titrate the sample.

V3 = ml. of ferric ammonium sulfate used to titrate the blank for TNT.

V4 = ml. of ferric ammonium sulfate used to titrate the sample for TNT.

N = Normality of ferric ammonium sulfate solution.

W = weight of sample.

4.3.1.6 Calcium silicate, Code No. 06002.-A weighed portion of 5.00 gm. of the sample shall be placed in a 500 ml. beaker and 50 ml. of acetone shall be added. The beaker shall be covered with a watch glass and placed on a steam bath and warmed. The lumps shall be broken up with a glass rod and the solution agitated occasionally by swirling. Decant the solution through a filtering crucible and treat the residue in the beaker with 20 ml. more of acetone. Filter the solution through the crucible. Transfer the insoluble residue completely to the crucible and wash it with acetone. The crucible and residue shall be dried in an oven at 110 degrees C. for 1 hour, cool in a desiccator and weigh. The percent calcium silicate shall be calculated as follows:

Percent calcium silicate = 100A W-MW

where:

A = the weight of the residue.

W = the weight of the sample.

M = percent moisture in material expressed as decimal (see 4.3.2).

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4.3.2 Moisture

- 4.3.2.1 Karl Fischer method (alternate method) Code No. 07001 Them moisture shall be determined in accordance with Standard FED-STL-791, Method 3253, except the sample shall be 8 to 10 grams.
 - 4.3.2.2 Conductimetric method Code No. 07002
 - 4.3.2.2.1 Apparatus.-The following apparatus is required:
- a. A conductivity cell having platinum electrodes coated with platinum black, and mounted in a glass case. The cell constant of the conductivity cell shall be approximately 0.1 reciprocal centimeter.
- b. A conductivity bridge, of the Wheatstone type, having a range from 10,000 to 100,000 ohms, and a precision of 2 percent or better.
 - c. A glass stirrer of the propeller type.
 - d. A stirring motor.
- e. A 200 milliliter (ml.) automatic pipette connected by means of a ground glass connection with a 5-gallon pyrex reservoir.
- f. A 50-ml. automatic burerte connected by means of a ground glass connection with a 5-gallon pyrex reservoir.
- g. A wide-mouthed glass titration bottle of approximately 400-ml. capacity equipped with a tight fitting ground glass stopper.
- h. A rubber stopper which fits the titration bottle, and which is provided with 3 holes, one for the conductivity cell, one for the stirrer, and one for the tip of the burette.
- 4.3.2.2.1.1 Assembly.—The apparatus shall be assembled as follows: The conductivity cell shall be inserted into the appropriate hole of the rubber stopper. A short piece of glass tubing shall be placed into another hole of the rubber stopper, to act as a sleeve for the shaft of the stirrer. The internal diameter of the glass tubing used shall be only slightly greater than the diameter of the shaft of the stirrer. The rubber stopper assembly shall be clamped to a suitable support. The shaft of the stirrer shall then be passed through the sleeve in the rubber stopper and attached to the stirring motor. The stirring motor shall be provided with a suitable support. The burette shall be set up so that the tip of the burette passes completely through the third hole in the rubber stopper. The electric leads from the conductivity cell shall be attached to the proper binding posts of the conductivity bridge.

4.3.2.2.2 Solutions.

- 4.3.2.2.2.1 Solution "A" (Acetic Acid-sulfuric acid).-A portion of 16.0 liters of approximately 99.9 percent glacial acetic acid shall be mixed with 85 ml. of approximately 96 sulfuric acid and 30 ml. of approximately 97.5 percent acetic anhydride in the 5 gallon pyrex reservoir, which is to be used with the 200 ml. automatic pipette. This solution shall have a blank titration value of not more than 3 ml. of solution B as determined in 4.3.2.2.2.3. If one blank titration of the solution exceeds 3 ml. of solution B, more acetic anhydride shall be added.
- 4.3.2.2.2.2 Solution "B" (acetic anhydride-acetic acid).A portion of 2.0 liters of approximately 97.4 percent acetic
 anhydride shall be mixed with 15.0 liters of approximately 99.9
 percent glacial acetic acid in the 5 gallon pyrex reservoir which
 is fitted with the 50 ml. automatic burette. This solution shall
 have a water equivalent value of approximately 0.02 gram (gm.) of
 water per ml., when standardized as specified in 4.3.2.2.2.3.
 The water equivalent value of the solution shall be adjusted if
 necessary, by adding more acetic anhydride or glacial acetic acid,
 the former increasing the value and the latter decreasing it.

4.3.2.2.3 Standardization of the solutions.

4.3.2.2.3.1 Preferred procedure.-Two hundred ml. of solution A shall be pipetted into the titration bottle. All necessary precautions shall be taken to minimize absorption of moisture from the atmosphere during this, and the subsequent operations. A weighed portion of approximately 0.5 gm. of water shall be added to the bottle, and the bottle attached to the rubber stopper assembly. The stirring motor shall then be started, and operated at such speed that the solid material (if any) remains in suspension, and the resistance of the solution shall be practically constant as measured with the conductivity bridge. The agitated solution shall be titrated with solution B 0.5 ml. portions being added at a time, and the resistance determined after each addition. As solution B is added, the resistance of the solution being titrated increases to a max. at the end-point and then decreases. The resistance values obtained shall be plotted on rectangular coordinates against the corresponding number of ml. of solution "B" added. A straight line shall be drawn through the two points on the plot just preceding the point of max. resistance value, and the line extended to interesect a similarly extended line drawn through the two points on the plot just

following the max. resistance value. The point of intersection of the two lines shall be considered as the end-point of the titration. The number of ml. of solution "B" corresponding to this end-point shall be recorded. A blank determination shall be made on a 200-ml. portion of solution A. The number of gm. of water equivalent to 1 ml. of solution B shall be calculated as follows.

 $E = \frac{W}{V - V}$

where:

E = gm. of water equivalent to a 1 ml. of solution B

W = gm. of water added to standardization

V = ml. of solution B required to titrate the specimen.

v = ml. of solution B required to titrate the blank.

- 4.3.2.2.3.2 Alternate procedure.—The following procedure for determination of the titration end-point may be used as an alternate to the procedure specified in 4.3.2.2.3.1. The progress of the titration shall be followed by adjusting the conductivity bridge reading, sufficiently above or below the actual resistance of the solution so that the shadow in the "Eye" appears as a hairline. The reading of the bridge shall be increased uniformly so that the appearance of the shadow remains constant through the titration. The end-point is shown in the eye" by a slight opening of the shadow if the reading of the bridge is just below the resistance of the solution, and by the fading or disappearance of the shadow if the reading of the bridge is just above the resistance of the solution.
- 4.3.2.2.3 Procedure.-A specimen shall be crushed to a particle size of approximately 3/4 in. or smaller. A weighed portion of approximately 50 gm. shall be transferred to the titration bottle. A 200 ml. portion of solution "A" shall be added, necessary precaution being taken to minimize absorption of moisture from the atmosphere by the solution during this operation. It shall be ascertained that none or the specimen remains on the ground surfaces, then the bottle shall be stoppered with the glass stopper, and the contents of the bottle agitated until all of the TNT is The glass stopper shall be removed and the bottle dissolved. attached immediately to the rubber stopper assembly. The solution shall be titrated and the number of ml. of solution "B" equivalent to the end-point of the titration determined in a manner similar to that used in the standardization of solution "B" as specified in 4.3.2.2.3. The percentage of moisture in the specimen shall be calculated as follows:

Percent moisture = 100E(V-:)

where .

V = ml, of solution "B" required to titrate the specimen. v = ml, of solution "B" required to titrate blank.

E = gm. of water equivalent to 1 ml. of solution "B"

W = gm. of specimen.

- 4.3.3 Determination of viscosity (Efflux method), Code No. 08001 An efflux viscosimeter shall be prepared in accordance with Drawing 81-3-148 or approved equal, approximately 500 gm. of the sample material shall be placed in a melt pot and the agitator started. The jacket of the melt pot shall be heated with steam at 10 pounds per square inch. No control of temperature need be maintained in the pot other than that obtained by controlling the steam pressure. The temperature of the water circulating through the jacket on the viscosimeter cone shall be thermostatically controlled at 85 degrees C. As the sample melts it will flow into the viscosimeter cone. The material in the cone shall be stirred by hand with a thermometer until the temperature of the entire sample is 85 degrees C. Stirring of the molten sample shall be continued with occasional vertical movement, until it is entirely free of lumps and there is no segregation of RDX. The temperature of the sample shall be adjusted to 85.0 degrees C. and then the thermometer and rubber stopper removed from the bottom of the cone to permit the molten sample to flow freely into a pan. The time required for the surface of the molten sample to fall from the tip of the upper marker to the tip of the lower marker shall be measured with a stop watch graduated in tenths of a second. The timing shall be started at the instant the upper indicating pointer pierces the surface of the molten sample and stopped when the surface is broken by the lower indicating pointer. This time interval is the efflux viscosity of the sample, and shall be recorded to the nearest tenth of a second.
- Insoluble particles, Code No. C9COl.-A 50 gm. portion of the sample shall be weighed in a 400 ml. beaker. One-hundred ml. of acetone shall be added and the beaker and contents heated on a steam bath until all the lumps are broken down and all soluble material is dissolved. The mixing shall be poured through a small US Standard Number 60 sieve complying with the requirements of Specification RR-S-366. Care should be taken to wash all the insoluble matter from the beaker with acetone. The residue on the sieve shall be washed with acetone to remove the RDX and dry the sieve. The particles retained shall be counted.

- 4.3.5 Form, Code No. 10001.-The form shall be determined visually.
 - 5. PREPARATION FOR DELIVERY
 - 5.1 Packing (see 6.1)
- 5.1.1 Level A.-Composition B4 shall be packed and marked in accordance with Drawing 7548644.
- 5.1.2 Level C.-Composition B4 shall be packed and marked in accordance with drawing 7548645.
 - 6. NOTES
- 6.1 Ordering data.-Procurement documents should specify the following:
 - a. title, number and date of this specification.
 - b. level of protection required.
 - 6.2 Inspection code numbers.-The five digit code numbers assigned to the inspection herein are to facilitate future data collection and analysis by the Government.
 - 6.3 Chromous Chloride solution.-Method for the preparation of 0.2N chromous chloride solution may be found in Picatinny Arsenal Technical Memorandum No. ACS-3-60 "Determination of Nitrogenous Compounds of Ordnance Interest by Chromous Chloride Reduction (1) Compound containing Nitro and Nitramine Groups" by Charles C. Jamison dated May 1960 which may be obtained from Picatinny Arsenal, Dover, N.J.

Notice.-When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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APPENDIX B

MIL-C-51077 (Ord)
Calcium Silicate, Technical

MILITARY SPECIFICATION

CALCIUM SILICATE, TECHNICAL

1. SCOPE

1.1 This specification covers only one type of calcium silicate, absorbent for use in explosives.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitiation for bids form a part of this specification to the extent specified herein.

SPECIFICATIONS.

MILITARY

UU-8-48 - Sacks, Shipping, Paper

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.

MIL-STD-109 - Inspection Terms and Definitions.-MIL-STD-129 - Marking for Shipment and Storage.

PUBLICATIONS

ORDHANCE CORPS

ORD-M603-11 - Procedures and Tables for Continuous Sampling by Attributes.

F8C: 6810

(Copies of specifications, standards, drawings and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer).

3. REQUIREMENTS

3.1 Chemical requirements.-The calcium silicate, absorbent shall conform to the chemical composition specified in Table I, when tested as specified in the applicable paragraph.

Table I Percent Applical					
Ingredient	Maximum (max.)	Minimum (min.)	paragraph		
Total volatile matter Total silicon as silicon	9.0		4.3.1		
dioxide (SiO ₂) dry basis	64.0	52.0	4.3.2		
Total calcium as calcium oxide (CaO) dry basis	32.0	23.0	4.3.3		
Sum of percent SiO2, CaO, and loss of weight on ignition, dry basis	••••	9 7. 0	4.3.4		
рH	9.0	7.5	4.3.7		

^{3.2} Physical requirements.-The calcium silicate, absorbent shall conform to the physical requirements specified in Table II, when tested as specified in the applicable paragraph.

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Procerty	Table II <u>Percent</u> <u>Min. Max.</u>	Applicable paragraph
Granulation No.		4.3.5
Retained on 140 mesh sieve	1.0	
Retained on 325 mesh sieve	6.0	
Meta-nitrotoluene absorption	550 gram (gm.)/ 100 gm	4.3.6
Grit**	0.01	4.3.8

**Note: The residue shall not produce a scratching noise or scratch the glass slide.

- 3.2.1 Color.-The calcium silicate, absorbent shall be white in color.
- 3.3 Workmanship.-All bags shall be dry and all bags, and the material contained therein, shall be free of dirt, oil, grease and other foreign material.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 General quality assurance provisions.—The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements. Reference shall be made to Standard MIL—STD—109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.
- 4.1.1 Contractor quality assurance system.—If the contractor desires to utilize a quality assurance system, which is at variance with the quality assurance provisions of 4.2.2 and 4.3 and other documents referenced herein,

he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection. (measuring and testing equipment), and provisions for control and disposition of non-conforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance provisions required by the provisions of 4.2.2. and 4.3 and the other documents referanced herein. The contractor shall not be restricted to the inspection station or the method of inspection listed in this specification provided than an equivalent control is included in the approved quality assurance procedure. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any changes to the written procedure that effects the degree of assurance required by this specification or other documents referenced herein.

- 4.1.2 <u>Submission of product</u>.-At the time the completed lot of product is submitted to the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product submitted:
- a. A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.

b. Quantity of product inspected.

c. Results obtained for all inspection performed

- d. Specification number and date, together with an identification and date of changes.
- e. Certificates of analysis on all material covered by referenced government specifications procured directly by the contractor.

f. Quantity of product in the lot.

g. Date submitted.

The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.1.3 Government verification. Using the contractor's written quality assurance procedure (see 4.1.1), this detail specification, and other contractual documents as a guide, the Government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with a. or b. as applicable, the decision being the re-

sponsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality assurance provisions of other specifications referenced in any of the contractual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be as directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejection, screening, re-sampling, re-instruction of the supplier's employees, or other appropriate action:

- a. Verification at the point of manufacture shall be accomplished at unscheduled intervals in accordance with 4.1.3.1 and 4.1.3.2.
- b. Verification at the point of delivery shall be in accordance with 4.1.3.2.
- 4.1.3.1 Surveillance. Surveillance shall include, but is not limited to:
- a. Observation of procedures concerning lot formation and identification.
- b. Observation of sampling procedures and application of acceptance criteria.
- c. Determination that all required examinations and tests are performed in accordance with the prescribed procedures of this specification, or approved equivalents thereto.
- d. Review of procedures for control and disposition of non-conforming material.
- 4.1.3.2 Product inspection. Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection by the Government shall be performed in order to determine that the product is of the quality required by this specification and that the contractor's records are reliable.
 - 4.2 Inspection provisions. -
- 4.2.1 Lot formation. A lot shall consist of one or more batches of calcium silicate, absorbent, produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each batch shall consist of that quantity of calcium silicate, absorbent that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous.
- 4.2.2 Examination. Sampling plans and procedures for the following classification of defects shall be in accordance with Standard MIL-STD-105. Continuous sampling plans, in accordance with Handbook ORD-M608-11 may be used if approved by the procuring activity. Also, at the option of the procuring activity, AQL's and sampling plans may be applied to the

individual characteristics listed using an AQL of 0.25 percent for each major defect and an AQL of 0.40 percent for each minor defect.

4.2.2.1 Paper shipping sack, prior to filling (see 5.1) .-

Categorie	Defects	Method of inspection
Critical:	None defined	
Major:	AQL 0.25 percent	
101.	Sack torn, cut or punctured	Visual
102.	Seam incomplete, or improperly	
	finished	Visual
Minor:	AQL 1.00 percent	
201.	Marking misleading or unidentifiable	Visual
202.	Evidence of moisture	Visual-Tactile
4.2.2.2	Paper shipping sack, after filling (see	5.1)
Categorie	<u>Defects</u>	Method of inspection
Critical:	None defined	
Major:	AQL 0.25 percent	
101	Clasura incomplete on immuonen	17:1

Minor: None defined.

4.2.3 Testing. -

- 4.2.3.1 Sampling for tests. A sample of three pounds shall be selected from each lot for tests. If any sample fails to comply with any of the requirements, the lot shall be rejected.
 - 4.3 Test methods and procedures. -
- 4.3.1 Determination of total volatile matter. A portion of approximaterly five gm. weighed to the nearest milligram (mg.) shall be transferred to a tared glass or aluminum dish. The specimen shall be spread loosely over the bottom of the dish. The dish and contents shall be heated in a drying oven maintained at 1050 + 5 degrees centigrade (°C.) for four hours. The dish shall then be removed and placed in a desiccator. When cool, the dish shall be weighed to the nearest mg. The procedure of heating cooling and weighing shall be repeated at hourly intervals until constant weight is reached or the sample gains weight. The percent of total volatile matter shall be calculated as follows:

Percent total volatile matter = (A-B) 100

where:

A = original weight of the specimen in gms.

B = final weight of the specimen in gms.

4.3.2 Determination of total silicon as SiO₂. - A portion of approximately 0.5 gm. weighed to the nearest 0.1 mg., shall be transferred to a 250 milliliter (ml.) beaker. A 25 ml. portion of concentrated hydrochloric acid shall be added and the mixture shall be carefully evaporated to dryness on a hot plate in the hood. The residue shall be baked for 30 minutes in an oven maintained at 1050 + 50C. At the end of this period, the beaker shall be removed and allowed to cool to room temperature. The residue shall be moistaned with three ml. of concentrated hydrochloric acid and then 50 ml. of water shall be added and the mixture heated to boiling. This mixture shall be filtered through a Whatman Number (No.) 42 paper, or equivalent. The beaker shall be rinsed alternately with three portions of hot water, 20 ml. each, and three portions, 2.0 ml. each of 1:20 hydrochloric acid. Each portion of wash water shall be filtered through the original filter paper. The filtrate shall be evaporated to dryness on a hot plate and the residue treated as above beginning with baking in the oven. The residue shall be allowed to cool and then ten ml. of concentrated hydrochloric acid shall be added and then evaporated to dryness on a hot plate. The residue shall be diluted with water, heated to boiling, filtered and washed as before. The filtrate and washings shall be reserved for the determination of total calcium as CaO. Both filter papers shall be placed in an ignited and tared platinum crucible. The papers shall be dried over a low flame and then permitted to char without flaming. The crucible shall be transferred to a muffle furnace maintained at 950° + 50°C. for one hour. The crucible shall then be removed and cooled to room temperature in a desiccator. The residue should be a powdery material ranging in color from a light gray to a white or off white. If the residue is darker than a light gray, it shall be moistened with concentrated sulfuric acid and then gently heated over a flame in the hood to evaporate the sulfuric acid....It shall be transferred to a muffle furnace and ashed as before for 15 minutes. The crucible shall be removed from the furnace and cooled in a desiccator and weighed. Hydrofluoric acid shall be cautiously added dropwise to the crucible until there is no further reaction and the residue is covered with hydrofluoric acid. Five drops of concentrated sulfuric acid shall be added and the crucible shall be carefully heated on a hot plate until fumes of sulfur trioxide are emitted. The residue shall be allowed to cool, and then five drops of hydrofluoric acid shall be added. The crucible shall be heated as before until fumes are no longer evolved. The crucible shall be transferred to a muffle furnace, maintained at 9500 + 500C., for onehalf hour. The crucible shall then be removed and placed in a desiccator and weighed when cool. The percent of total silicon as SiO2 shall be calculated as follows:

Percent total silicon as $SiO_2 = \frac{(A-B) 100}{S (1 - \frac{7}{100})}$

wheres

A = weight of crucible in gms. with residue prior to addition of hydrofluoric acid.

B = weight of crucible in gms. after evaporation with hydrofluoric and sulfuric acids.

S = weight of the specimen in gms.

V = percent total volatile matter (see 4.3.1)

4.3.3 Determination of total calcium as CaO. - The filtrate and washings reserved from the SiO2 determination shall be quantitatively transferred to a 400 ml. beaker. The volume shall be adjusted to approximately 200 ml. and three drops of methyl red indicator added. Five ml. of concentrated hydrochloric acid shall be added, followed by 75 ml. of four percent ammonium oxalate. The solution shall be heated to 75° ± 5°C., and 1-1 ammonium hydroxide added with stirring until just alkaline. The solution shall be allowed to stand at room temperature for one hour with occasional stirring and then filtered through Whatman No. 42 paper, or equal. The precipitate shall be washed four to five times with cold 0.1 percent ammonium oxalate solution. The filtrate shall be discarded. Small portions of hot 1:4 hydrochloric acid, making a total of approximately 50 ml. shall be poured on the precipitate. The filtrate shall be caught in a 400 ml. beaker. The filter shall be washed with several small portions of 1:100 hydrochloric acid. The filtrate shall then be diluted to A 50 rd. portion of four percent ammonium oxalate solution shall be adde?. The solution shall be heated to incipient boiling. Calcium oxalate shall be precipitated by adding 1:1 ammonium hydroxide until the solution is just alkaline to methyl red and then by adding five ml. in excess. The solution shall be allowed to stand for one hour and then filtered through a Whatman No. 42 filter or equal. The beaker and the precipitate shall be washed with small portions of ice cold water until the washings are free of chloride ions. The minimal amount of water required shall be used. The filter paper and precipitate shall be returned to the beaker in which the precipitation took place. The precipitate shall be dissolved by adding 100 ml. of warm 1:10 sulfuric acid to the beaker, using a stirring rod to facilitate solution while warming. The solution shall be heated to 80°C. and then titrated at this temperature with standard 0.1 normal (N) potassium permanganate solution. The percent of total calcium as Can shall be calculated as follows:

Percent total calcium as
$$CaO = \frac{A \times N \times 2.804}{S(1 - \frac{V}{100})}$$

where:

A = volume in ml. of standard permanganate solution used in titration.

N = normality of the standard permanganate solution.

S = weight of the specimen in gms.

V = percent total volatile (see 4.3.1).

4.3.4 Sum of percents SiO_2 , CaO_3 , and loss in weight on ignition.— The loss in weight on ignition shall be determined as follows: A 2.0 gm. portion of the sample, weighed to the nearest 0.1 mg., shall be transferred to a previously ignited and tared crucible. The crucible and contents shall be gently ignited over a small flame in the hood. After the contents have been thoroughly charred, or fumes are no longer emitted, the crucible shall be more strongly ignited. After such treatment for ten minutes, the crucible and its contents shall be transferred to a muffle furnace, maintained at $980^{\circ} \pm 15^{\circ}C$., for one hour. At the end of this period the crucible shall be removed and placed in a desiccator. When cool, the crucible shall be weighed. The percent loss in weight on ignition shall be calculated as follows:

Percent loss in weight on ignition =
$$\frac{(A-B)\ 100}{A\ (1-\frac{V}{100})}$$

where:

A = weight of original specimen in gms.

B = weight of specimen in gms. after ignition.

V = percent total volatile matter (see 4.3.1).

The sum of percents SiO_2 , CaO, and loss in weight on ignition shall be calculated as follows:

Sum of percents SiO2, CaO and loss in weight on ignition = A+B+C

wheres

 $A = percent SiO_2$ (see 4.3.2).

B = percent CaO. (see 4.3.3).

C = percent loss in weight on ignition (see 4.3.4).

4.3.5 Granulation. - A 10.0 gm. portion of the material, weighed to the nearest 0.01 gm. on a torsion balance or equivalent, shall be transferre to a 250 ml. beaker. Slowly and carefully, 100 ml. of water shall be added down the side of the beaker so as not to disturb the powder. The mixture shall be permitted to stand undisturbed until the powder has been completely wetted. The slurry shall be transferred with the aid of a gentle stream of water, to a No. 140 United States (U.S.) Standard sieve nested over a No. 325 U.S. Standard sieve seated in a large casserole or dish. The balance of the residue n the beaker shall be transferred quantitatively to the No. 140 sieve, by means of small washes of water. When the entire slurry has been washed onto the No. 140 sieve, the washing shall be continued using a fine camel's hair brush to manipulate the slurry on the sieve. When the water passing through the sieve is clear, the sieve shall be removed and dried in an oven, maintained at 1050 + 50C. for four hours. The sieve shall then be removed, cooled to room temperature and weighed. This residue shall be retained for the grit determination. The gain in weight is due to particles too large to pass through the meshes of the sieve. In the interim, while the No. 140 sieve is drying, the slurry remaining on the No. 325 sieve shall be washed using the same procedure as for the No. 140 sieve. The No. 325 sieve shall be dried as the No. 140. This residue shall also be retained for the grit determination. The percent by weight retained on the No. 140 and No. 325 sieves shall be calculated as follows:

a. Percent retained on No. 140 sieve = $\frac{(A-B) \times 100}{S}$

where:

A = weight of No. 140 sieve and residue in gms.

B = weight of No. 140 sieve in gms.

S = weight of specimen in gms.

b. Percent retained on No. 325 sieve = $\frac{(C-D) \times 100}{S}$

where:

C = weight of No. 325 sieve and residue in gms.

D = weight of No. 325 sieve in gms.

S = weight of specimen in gms.

4.3.6 Meta-nitrotoluene absorption. - A 5.0 gm. specimen weighed to the nearest 0.01 gm. shall be transferred to a 500 ml. casserole and meta-nitrotoluene shall be added from a burette at the rate of 0.15 to 0.20 ml. per second. During the addition of the liquid, the mixture shall be thoroughly stirred with a four inch blade spatula. The flow from the burette shall be stopped when the mixture becomes slightly fluid and the

liquid shall be added drop by drop until the end point is reached. The end point shall be reached when the mixture is sufficiently fluid to drop off the end of the spatula in individual drops when held in a vertical position. This test shall be completed within 10 minutes (max.).

- 4.3.6.1 <u>Calculation</u>.- Multiply the volume delivered by 23.2 and report as gms. meta-nitrotoluene absorbed per 100 gms. of sample.
- 4.3.7 pH.- A ten gm. portion of the sample shall be weighed to the nearest 0.01 gm. on a torsion balance or equivalent scale, and placed in a 250 ml. beaker. A 100 ml. portion of distilled water shall be added slowly down the side of the beaker. The mixture shall be stirred with a glass stirring rod and then allowed to stand for ten minutes. The solution shall then be filtered with suction through a Buchner funnel using a previously washed Whatman No. 1 filter paper or equivalent. The first ten ml. of the filtrate shall be discarded. The pH of the filtrate shall be taken using a Beckman glass electrode pH meter, or equivalent. (The pH of the distilled water employed in this determination, should be between 6.0 and 7.0).
- 4.3.8 <u>Grit.</u>— The residue obtained from the gramulation determination in paragraph 4.3.5 shall be transferred to a tared weighing bottle, dried at 100° to 105°C. for one hour, cooled in a desiccator and weighed. Calculate the weight of residue to percent grit. After performing the test place the material between two glass slides. Rub the slides together to determine the presence of grit by scratching noise and scratches on the glass slide.

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging .-

- 5.1.1 <u>Level A.-</u> The calcium silicate absorbent shall be packaged in conformance with Specification UU-S-48, sack construction No. 2X of table I.
- 5.2 Marking.- Marking shall be in accordance with Standard MIL-STD-

6. NOTES

- 6.1 Ordering data. Procurement documents should specify the title, number, date of this specification, and quantity required.
- 6.2 Intended use. The calcium silicate is intended for use in explosive compositions containing Trinitrotoluene (TNT) (to prevent exudation of TNT).

Notice. - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation or conveying any rights or permission to manufacture, use, or sell any patented invention that in any way be related thereto.

Custodian:

Army - Ordnance Corps

Preparing Activity: Army - Ordnance Corps

GPG 617877

APPENDIX C

MIL-L-20336A
Loading, Assembling and Packing
of
High Explosive Shell
with
Cast Trinitrotoluene (TNT), and Cast Composition B

MIL-L-20336A

14 DECEMBER 1954

SUPERSEDING MIL-L-20226 27 NOVEMBER 1951

MILITARY SPECIFICATION

LOADING, ASSEMBLING, AND PACKING OF HIGH EXPLOSIVE SHELL WITH CAST TRINITROTOLUENE (TNT), AND CAST COMPOSITION B

This specification has been approved by the Department of Defense for use of the Departments of the Army, the Navy, and the Air Force.

1. SCOPE

1.1 This specification covers the loading, assembling, and packing of the following classes of high explosive shell with cast trinitrotoluene and cast Composition B:

Class A. Separate-loaded, issued unfuzed

Class B. Separate-loaded; issued fuzed

Class C. Fixed, semi-fixed, and mortar rounds

2. APPLICABLE DOCUMENTS

2.1 The following specifications, and publications, together with the Ordnance Corps loading drawing pertaining to the shell under contract (see 6.1), and all drawings and specifications referenced thereon, of the issue in effect on date of invitation for bids, form a part of this specification:

SPECIFICATIONS

MILITARY

MIL-G-2550 — General Specification For Ammunition Except Small Arms Ammunition

MIL-R-11470— Radiographic Inspection; Qualification of Equipment, Operators and Procedures

PUBLICATIONS ---

ORDNANCE CORPS

ORD-SIP-M11-4 — Shell, Separate Loading, HE Nose Fuzed, Loaded

ORD-SIP-M11-27 — Shell, HE. 280MM T122, T122E3, and T122E4. Loaded

ORD-SIP-M11-501— Loading of HE
Shell With Cast TNT and Cast Composition B

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring agency or as directed by the contracting officer.)

3. REQUIREMENTS

3.1 Material. ..

3.1.1 Material and parts shall be in accordance with applicable specifications and drawings.

3.1.2 The explosive shall comply with all requirements specified on applicable drawings and specifications.

3.1.3 Clean scrap produced in regular operations may be remelted and reused provided that it complies with the requirements for the grade of high explosive specified, except for form and viscosity. Scrap from floor sweepings, ventilators, and suction appara-

TABLE I.

Califer of shell	Designation	Type of fuse well	Segment \ (mehen)	Segment C approx (inches)	Segment D (inches)
57-mm	M306A1	Shallow	0.5	3.2	0.0
60-mm	T21E2	Shallow	1.0	2.5	0.0
75-mm	M302A1	Shallow	0.5	2.5	0.0
75-mm	M18	Shallow	2.0	2.5	0.0
75-mm	M48	Deep	1.5	4.3	2.0
75-mm	T50E2	Deep	2.6	2.9	2.5
76-mm	M42A1	Shallow	2.0	3.7	0.0
76-mm	T64	Shallow	2.0	2.5	0.0
81-mm	M43A1B1	Shallow	0.5	2.5	0.0
81-mm	M56A1	Shallow	1.0	2.5	0.0
81-mm	T28E7	Shallow	1.0	-2.5	0.0
90-mm	M71	Shallow	2.5	2.5	0.0
90-mm	M71	Drep	2.0	- 2.5	2.0
90-mm	T91	Shallow	2.0	2.5	0.0
105-mm	Mi	Shallow	2.5	2.5	0.0
105-mm	Mi	Deep	2.5	2.5	2.0
105-mm	· M323	Deep	1.0	~ 2.5	2.0
105-mm	T53E2	Shallow	1.5	2.5	0.0
4.2-in.	M3A1	Deep	3.0	2.5	2.0
4.2-in.	M329	Deep	1.5	2.5	2.0
120-mm	M73	Shallow	2.5	.2.5	0.0
120-mm	M73	Deep	2.5	2.5	2.0
120-mm	T15E3	Shallow	3.0	2.5	0.0
155-mm	M101	Shallow	3.5	2.5	0.0
155-mm	31101	Deep	3.5	2.5	2.0
155-mm	M107	Shallow	2.5	2.5	0.0
155-mm	M107	Deep	2.5	2.5	2.0
8-in.	M103	Shallow	2.5	2.5	0.0
8-in.	M106	Shallow	3.0	2.5	0.0
8-in.	M106	Deep	3.0	2.5	2.0
240-mm	- M114 -	Shallow	- 3.0	- 2.5	0.0
240-mm	M114	Deep	3.0	2.5	2.0
280-mm	T122E3	Deep	3.5	2.5	2.0

tus shall not be used. (Clean scrap is defined as scrap formed in kettles, pails, risers and loading machines used in the pouring and cooling operations.)

3.2 Charge.

3.2.1 Specific gravity of the charge.—The minimum overall specific gravity of the charge when determined as specified in 4.4 shall be as follows:

TNT	1.55
Composition B	1,62

3.2.2 Segments.

3.2.2.1 Definitions of segments.—For purposes of inspection and determination of cavities, the charge shall be divided into segments defined as follows:

Segment A shall be defined as the distance from the inside of the rear of the shell to the rear boundary of Segment B.

Segment B shall be defined as the distance from the forward boundary of Segment A to the rear boundary of Segment. C. If this distance is less than 2.5 inches, it shall be considered as 0.0

Segment C shall be defined as the distance from 2.5 inches below the base of the fuze well to the base of the fuze well. When Segment B is less than 2.5 inches, Segment C is the distance from the forward boundary of Segment A to the base of the fuze well.

Segment D (applicable only for charges containing a deep fuze well) shall be defined as extending from the forward boundary of Segment C. i.e. base of the deep fuze well, to a point 3.0 inches from the nose of the shell body. However, for the T50E2 HE Shell, the distance shall be 2.5 inches from the nose of the shell body instead of the 3.0 inches.

3.2.2.2 Dimensions of segments.—The dimensions of the segments in each of the shell shall be in accordance with Table I.

3.2.3 Cavities.

3.2.3.1 Cavities having a projected length of $\frac{1}{12}$ inch or less shall be disregarded. Cavities within the explosive charge shall not exceed the requirements specified in Table II when determined in accordance with 4.5.

3.2.3.2 Cavities in the base of a shallow fuze well shall not be cause for rejection pro-

TABLE II.

	Segment ,\	Segment B	Segment C	Segmen D
Sum of projected areas of the cavi- ties, excluding pipes, cracks and annular rings (sq. in.).	*3/n4	*	3%	1/2
Projected length of any cavity, exclud- ing pipes, cracks and annular rings (in.).	%	⅓	14	9 %
Piping cavities max, projected area (sq. in.).	0	14	1/4	0
Piping cavities, max. projected width (in.).	0	14	34	0
Cracks, max. projected width (in.),	1/1/2	142	142	142
Annual rings, max, projected width (in.).	0	0	*4	0

^{*} If the length of the largest cas.ty is 1/16 inch or less, the total projected area may be 1/20 square inch.

vided the sum of the areas of the cavities is not greater than 80 percent of the area of the fuze well base and no individual point extends more than ½ inch below the maximum depth of the fuze well permitted by the drawing. Cavities in the base of a deep fuze well (shell with a supplementary charge) shall not be cause for rejection provided the sum of the areas of the cavities in not greater than 25 percent of the area of the fuze well base and no individual point extends more than ¼ inch below the maximum depth of fuze well permitted by the drawing.

3.2.3.3 Porous areas.—Porous areas shall be treated as cavities, and shall be subject to the restrictions placed upon cavities, except that 80 percent of the projected length and 80 percent of the projected area shall be considered for acceptance purposes.

3.2.3.4 Cracked charges.—Not more than two transverse cracks shall be permitted in any charge, and not more than one crack shall be permitted in Segment A.

3.2.3.5 Surface cavities.—Pinpoint cavities on the explosive charge adjacent to the shell wall shall be permitted for mortar shell.

3.3 Loading of high explosive.

3.3.1 The mating surfaces of threads shall be free of caked explosive, and all threads shall be free of foreign material. If a chemical cleaner is required, no material other than acetone shall be used. Care should be taken to prevent acetone from coming into contact with the charge and becoming entrapped in the shell.

3.3.2 When TNT shows definite frothing tendencies (which may cause production of unsatisfactory charges) approval may be obtained from the responsible procurement agency for the use of authorized materials (such as 0.1 percent maximum of sorbitan trioleate, Specification JAN-S-547) for prevention of frothing.

3.3.3 Each shell, immediately prior to pouring, shall be inspected for foreign ma-

MIL-L-20336A

terial in the shell cavity, and each shell interior shall be free of foreign material.

- 3.3.4 Solid explosive shall not be present in the shell before loading or be added to the shell during pouring or cooling.
- 3.4 Coating.— Shell having the interior well imperfectly coated shall not be loaded.
- 3.5 The interior surface of the shell shall be dry when the high explosive is poured into the shell.

3.6 Workmanship.

- 3.6.1 The loaded shell shall be free of chips, dirt, grease, rust and other foreign material and shall be painted as required by the shell drawing.
- 3.6.2 Care shall be exercised, at all times so that the rotating band of each shell will not be damaged by being nicked or burred.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Lot.—A lot shall consist of all shell of the same designation loaded with cast trinitrotoluene, or cast Composition B, by an unchanged process by one loading plant. Each lot of Class A and Class B shell (see 6.2) shall contain:—
 - (a) Loaded shell of one weight zone when weight zoning is required.
 - (b) Supplementary charges from any plant, made in accordance with the same drawing and same drawing revision when supplementary charges are required.
 - (c) Fuze well cups from any plant, made in accordance with the same drawing, same drawing revision, same specification and same specification revision when fuze well cups are required.
 - (d) Shell metal parts of one interfix lot number made by one manufacturer.

- (e) Loaded point detonating fuzes (where applicable) from not more than one manufacturer of one interfix lot number.
- (f) Loaded time fuzes (where applicable) of the powder train type from not more than one manufacturer of one interfix lot number.
- (g) Loaded mechanical time fuzes
 (where applicable) from not more
 than one manufacturer of one interfix lot number, or mechanical
 time fuzes from not more than
 one grand lot made in accordance
 with the same drawing, same
 drawing revision, same specification and same specification revision.
- 4.2 Sampling.—Number of samples, acceptance and rejection criteria used for determination of lot acceptance for tests as specified in 4.4 and 4.5 shall be in accordance with Standard Inspection Procedure ORD—SIP-M11-501.
- 4.3 Inspection. Inspection shall be as specified in Specification MIL-G-2550. In addition for Class A and B shell, inspection shall be in accordance with Standard Inspection Procedure ORD-SIP-M11-4, or ORD-SIP-M11-27, as applicable.
- 4.4. Determination of specific gravity.—
 The inspector shall select and identify sample shell and the specific gravity of the charge in each sample shall be determined as follows:

Each sample shell shall be weighed empty (without explosive) (We). Each sample shell shall be filled with water and weighed (Ww). Each shell shall be emptied and thoroughly dried. The sample shell shall be placed in the loading line and shall be filled under conditions identical in all respects with those employed in filling regular shell. Each sample shell filled with explosive and having a finished fuze well, shall be weighed (Wt) and the same shell reweighed after filling the

fuze well with water (Wf). The water shall be emptied and the well thoroughly dried. The specific gravity shall be calculated as follows to determine compliance with 3.2.1.

Specific gravity = $\frac{Wt - We}{(Wm - We) - (Wf - Wt)}$ where:

We = weight of empty shell

Ww = weight of water-filled shell

Wt = weight of loaded shell with fin-

ished fuze well

Wf = weight of loaded shell with water-filled fuze well

4.5 Determination of cavities. — Sample shell shall be selected and subjected to examination by a visual inspection of the fuze well and by x-ray examination. The x-ray beam shall form a 90-degree angle with the vertical axis of the shell at a point within 2.0 inches of the base end of the charge. Examination of the x-ray negative shall be made to determine compliance with 3.2.3.

Note. Radiographic equipment, operators and procedures shall be qualified in accordance with Specification MIL-R-11470. In addition the sensitivity of x-ray technique shall be 2.0 percent, and the photographic density of the film used shall be no less than 1.25, as determined in accordance with 4.6.

- 4.6 Check of sensitivity of x-ray technique, and photograp, ic density of film.
- 1.6.1 The sensitivity of x-ray technique shall be determined, at intervals prescribed by the inspector, by means of a penetrameter fabricated as specified in 4.6.2. The penetrameter shall be placed on the side of the shell away from the film (source side). Observation shall be made of the image of the penetrameter on the x-ray film. The sensitivity of x-ray technique shall be considered satisfactory when the image of the penetrameter, including the hole in the center, is sharply defined on the x-ray film.
- 4.6.2 Fabrication of the penetrameter. The penetrameter shall be made of material equal to the combined metal plus explosive filler density and absorption factor at 1000

KV, and shall be of a thickness equal to 2.0 percent to the total equivalent thickness (metal plus charge) at the thickest part to be examined. The penetrameter shall have a hole in its center, the diameter of which shall be equal to the thickness of penetrameter.

4.6.3 Check of photographic density of film.—The photographic density of the film shall be determined on the film in a region of sound charge plus metal corresponding to that portion of the material which is of the greatest combined thickness to be examined on the negative. To judge densities, it is recommended that standards of radiographic densities be used (see 6.3). Comparison of film with standards may be made visually; however the use of a densitometer is recommended.

5. PREPARATION FOR DELIVERY

- 5.1 Packing, labeling and marking.—Packing, labeling and marking shall be as specified on the applicable drawing.
- 5.2 Data cards. Data card information shall be as specified in Specification MIL—G-2550.

6. NOTES

- 6.1 Ordering data. Procurement documents should specify the following:
 - (a) Title and date of this specification.
 - (b) Drawing required for shell under contract (see 2.1).
- 6.2 Necessary information on lot content on Class C shell may be obtained from the detail complete round specification.
- 6.3 "American Standard For Diffuse Transmission Density", published by American Standards Association, 70 East 40th Street, New York has been found satisfactory as a source of information on film densities.

MII-I-20336A

Notice.—This specification, together with specifications and drawings pertaining to it and bearing a "Notice" of similar restrictions, is intended for use only in connection with procurement by the United States Government, and shall not be reproduced either wholly or in part, except when authorized in connection with Government procurement, nor be

used for any other purpose except when specifically authorized by the Chief of Ordnance.

Custodians:

Army-Ordnance Corps Navy-Bureau of Ordnance.

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ECO NO

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SHEET 2 OF SHEETS

DATE

12 Dec 1956

ENGINEERING CHANGE ORDER

SUBJECT: LOADING, ASSEMILLING, AND PACKING OF HIGH EXPLOSIVE STIELL WITH CAST TRINITROPOLUTINE (THT) AND CAST COMPO-

SITION B

Segment A (inches)	2.0
Segment C approx. (inches)2.5	3.7
Seament D (notices) 0.0	0.0"

Page 2, Table I, under "Designation" heading, last item. -Substitute: "T122" in place of "T122E3".

Page 2, Table I, under "Designation" heading, item 8. - Substitute: "M352" in place of "T64".

Page 2, .Table I, under "Designation" heading, item 11. -Substitute: "M352" in place of "T28E7".

Page 2, Table I, under "Sc. ment C approx. (inches)" heading. -Substitute: "5.0" in place of "2.5" for 280MM, T122 Shell.

Page 2, paragraph 3.2.1. -Not applicable.

Page 2, para raph 3, 2, 2, 1, subpara graph on Segment C, sentence 2, -Add: "Except if otherwise defined in Table 1".

Page 3, paragraph 3. 2. 3. 2, sentence 2, -Substitute:

"cavities in the base of a deep fuze well (shell with a supplementary charge) shall not be cause for rejection provided the sum of the areas of the cavities is not greater than 60 percent of the area of the fuze well base and no individual point extends more than 1/4 inch below the maximum depth of fuze well permitted by the drawing, and provided no point extends above the flat surface on which the liner would rest".

Page 3, paragraph 3, 2, 3, 4, -Ad h

"Cracks will be permitted in Segment C of the 280MM, T122 Shell".

*Page 5, paragraph 1.6, 2, -Substitute the following:

"4.6.2 Fabricatio, and location of the penetrameter. Unless otherwise specified, the penetrameter shall be made of steel having an approximate specific gravity of 7.8. The thickness of the penetrameter shall be based on a combination of both charge and steel thicknesses and shall be equal to 2.0 percent of the maximum equivalent thickness (Fe). The equivalent thickness (Te), is the sum of the steel thickness (2Tw), and the charge thickness (Re) equated to steel thickness (Te x 1.6/7.8). The penetrameter shall have a holo, the diameter of which is twice the thickness of the penetrameter. For further details on construction of the penetrameter see Fig. 2 of Specification MIL-R-11471. On artillery shell, the maximum equivalent thickness will generally occur slightly forward of the lase of the charge cavity. The actual thicknesses may be determined from a radiograph of the metal parts. The penetrameter thickness shall be determined from Fig. 1."

ORDNANCE CORPS ORDNANCE AMMUNITION COMMAND JOLIET, ILLINOIS



ECO NO 129767 SANR

DATE

12 Dec 1956

ENGINEERING CHANGE ORDER

LOADING, ASSEMBLING, AND PACKING OF HIGH EXPLOSIVE SHELL WITH CAST TRINITROTOLUENE (TNT) AND CAST COMPO SUBJECT: SITION B

SHEET 3 OF 4 SHEETS

Page 6, section 6. -Add the following:

"6.4 Except when shell are examined by x-ray, the specific-gravity of the charge shall be determined as specified in 4.4 and reported for information at least once per shift. Corrective action shall be taken if the specific gravity is below the following minimum value

> TNT 1.55 Composition B 1.62"

*Add attached figure 1.

(Contract Application for Army Ordnance-Ammunition: 5 on A and B, 1 on C)

This ECO-ANR supersedes ECO-128362-S ANR, dated 30 January 1956 to Specification MIL-L-20336A.

*Asterisk indicates changes in addition to or differing from ECO-128362-S ANR.

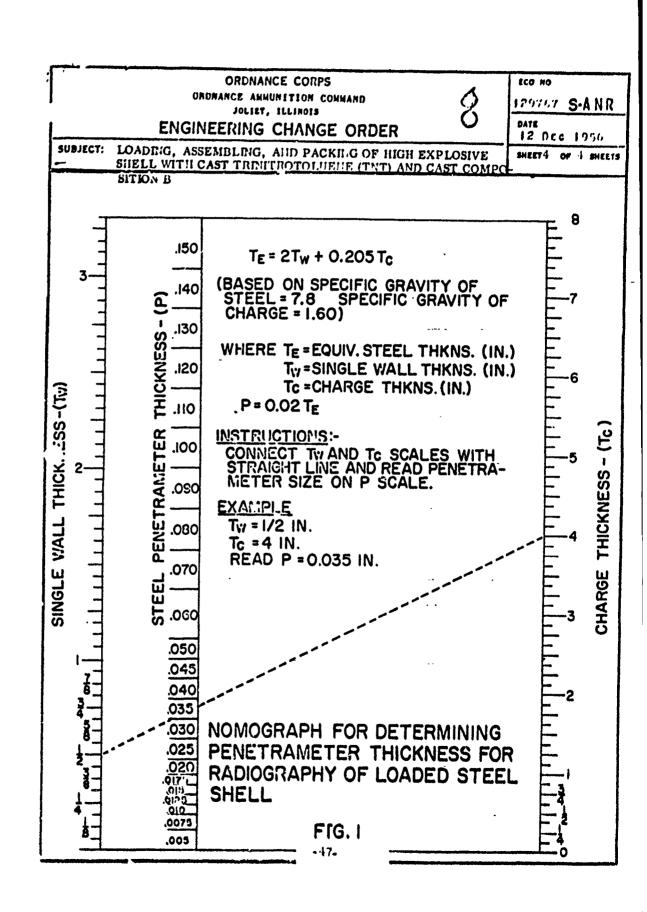


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